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IMPLEMENTATIVE INVESTIGATION OF PRACTICAL METHODS OF
ACCELERATED SINTERING OF REINFORCED CERAMIC

To: U. S. Energy, Inc., et al.

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COMPARATIVE INVESTIGATION OF VARIOUS METHODS OF
ACTIVATED SINTERING OF REDUCED IRON

BY: R. A. Andriyevskiy, et. al.

English Report

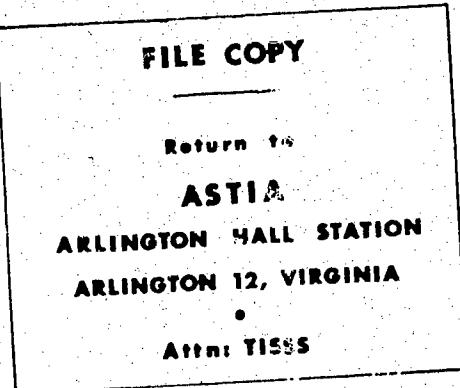
SOURCE: Zopovidi Akademii Nauk Ukrainskoy RSR, Nr. 4,
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COMPARATIVE INVESTIGATION OF VARIOUS
METHODS OF ACTIVATED SINTERING OF
REDUCED IRON

Porivnyalne Doslidznennya Riznikh Metodiv
Aktivovanogo Spikannya Vidnovlenogo Zaliza

In several works, (for example, /1-3/) were discussed different methods of activated sintering, which are designed to intensify sintering by the way of chemical reactions of oxidation-reduction, disassocation and other, or with the help of different physical methods,- action of ultra-sound, for example. It is time to determine the most effective method of activation. Tests were done on toroidal samples from reduced powder of iron of the mark APZHM. The following process of sintering were tested: I - Sintering in dry hydrogen(0,022% of H₂O); II - sintering in moist hydrogen(2% H₂O); III - humidity of hydrogen 10%; IV - sintering of pre-oxidized briquettes (1% O₂); V - cyclic sintering (3 times with H₂O steam, 7 times with H₂); VI - sintering in atmosphere of H₂ + HCl; VII - sintering while covered by Al₂O₃ + 0.1% NH₄Cl; IX - sintering while covered by Al₂O₃ + 0.1% NH₄F.

Heating and cooling (6—10 times) was realized in dry hydrogen; the other 20 expositions in sintering were conducted under conditions II, III, V, VI, VII and in atmosphere of dry hydrogen. The temperature and duration of expositions were alike for all tests — 2 hrs, 1200 ± 10° c.

The figure shows the influence of specific gravity of the samples which were sintered under different conditions on coercive force Hc, maximum magnetic penetration (M), magnetic induction in the field of 15 e (B₁₅) and magnetic residual induction Br. Magnetic properties were chosen as they are considered to be the ~~most~~ most sensitive to the form of the pores, and they were measured at the standard ballistic installation/4/. The results of measurements of the (feeded) surface of open pores S¹ and change in chemical content are given in the following table.

CONDITIONS OF SINTERING	S, m^2/gr	C, %	Mn, %
Compressed briquetts	0,20	0,10	0,32
I. Sintering in H_2	0,15	0,02	0,30
II. III Sintering in moist H_2	0,15	0,01	0,31
IV. Sintering of oxidized samples	0,12	0,02	0,30
V. Cyclic sintering(3 times H_2 0.7 times H_2)	0,13	0,01	0,30
VI. Sintering in $\text{H}_2 + \text{HCl}$	0,07	0,01	0,03
VII. Sintering of oxidized samples in $\text{H}_2 + \text{HCl}$	0,065	0,01	0,03
VIII. Sintering with NH_4Cl	0,10	0,06	0,16
IX. Sintering with NH_4F	0,13	0,07	0,30

The investigation of microstructures has not shown a noticeable difference in the dimensions of the graphs of samples, which were sintered at the different conditions. As it was shown by the results of the investigation, the longest influence on the values of " and Hc has been at conditions VI and VII, that is with application of hydrogen chloride.

- 1) The measurements were conducted according to the method of B. V. Deryagin/5/, perfected by us for experimentation with compressed briquetts (dia 10 mm, h = 5 mm), which were sintered under conditions I-IX during one hour at 1200°C. The number of open pores has been about 25%.

Density gr/cm³

Influence of densities on magnetic properties of samples which were sintered at different conditions of activated sintering.

- X — condition I, sintering in H₂
- ♀ — condition II, sintering in moist H₂(\approx of H₂O)
- + — CONDITION III, sintering in moist H₂(10% of H₂O)
- — condition IV, sintering of oxidized samples
- □ — CONDITION V, ~~sintering~~ cyclic sintering
- A — condition VI, sintering in H₂ + HCl
- ▲ — condition VII, sintering of oxidized samples in H₂ + HCl
- ○ — condition VIII, sintering with Al₂O₃ + 0.1% NH₄Cl
- — condition IX, sintering with Al₂O₃ + 0.1% NH₄F

The mechanism of HCl is an intensification of processes changing form of the surface gravity and refining chemical content (see table). The presence of HCl results from the formation on the edges of the pore of FeCl₂ which is evaporated and reduced by hydrogen. The atoms of iron so formed are concentrated in places of the minimum content of free energy (cavity on the surface particles, joints of particles) and hydrogen chloride

again reacts with iron on the action joints of surfaces and etc. By this process the surface ^{of} grains is smothered; the analogous process occurs also with admixture of NH_4Cl and NH_4F (for the last case the iron fluoride is fusing at 1100°C) /1/. The surface of HCl is responsible for the formation of manganese chloride, which evaporates easily, and its contents in samples is diminishing.

This large spheriod form of pores reduces the distinguishing influence of oppositely directed poles, which are forming on the protusions of the surface pores. Admixtures also influence magnetic properties. As far as during sintering under conditions I — VIII the carbon content, which must greatly influence magnetic properties,/6/, remains almost unchanged (0,01—0,02%), then the improvement of H_1 and H_c at the sintering with adding HCl to atmosphere can be attributed to the influence of surfaces pores, because manganese affects H_1 and H_c in considerably lower degree than carbon. So, according to emperic formulas /6/ 0.27% of Mn changes H_c or about 0.1 erg., but in our case, we have a much greater difference—0.3-0.6 erg.

In the treatise /7/ the theory on the dependence of dielectric and magnetic penetration on the density with accounting for the form of pores(spherical, disc-shaped, acicular) has been developed; our results clearly support its conception.

(1) Sensitivity. Sensitivity of H_1 and H_c to the form of pores /6/ and influence of refining has also been noted in investigations /8, 9/.

The role of refining (by carbon) is evident when the surfaces sintered at conditions V and IX are compared. The active/surface of pores was $0.13 \text{ m}^2/\text{gr}$, but high content of carbon during the sintering with NH_4F (the note on the container explains it is as a restricted passage to hydrogen) is responsible for low values of magnetic characteristics.

I) In monograph (11) we tried to differentiate the influence of the surface pores conditions on the change of physic-mechanical properties during isothermal sintering of iron. The result had been considered slightly higher due to incomplete accounting of density influence and partial refining.

According emperic formulas /6/ 0.06% of C change Hc on about 0.2 erg, but in our case the difference was about 0.4 erg, probably due to additional negative influences of iron fluoride forming during the process.

At the sintering with NH_4Cl the high content of carbon was also observed, but small active(feeding) surfaces (and particularly the smaller content of manganese) probably compensate for this fact. Sintering with NH_4Cl and NH_4F can be recommended highly for the construction materials, as the small admixtures influence mechanical properties considerably less than they influence magnetic properties and effect of smothering of pores expresses in full scale /1-3/. Sintering at conditions II and V less than at conditions VII affect the intensification of changes in magnetic properties. The results, as a rule, correspond to certain disintegration, especially for condition V in internal of small densities, which can be explained by specific characters of oxidation of ~~xxxxxx~~ porous bodies. Moistness of hydrogen contributes only to the decrease in Hc. Magnetic and residual induction depends basically on the density of porous bodies, although this ~~xxxxx~~ process is somewhat dependant on certain dispersion.

The admixtures of hydrogen chloride into the atmosphere of sintering favorably affects the properties of metallo-ceramics bodies, but the presence of HCl diminishes anticorrosive properties of samples and requires introduction of special measures of the safety technique. Therefore, the further investigation is necessary to determine an ~~optimum~~ optimal ~~greatest~~ quantity of HCl. According to /10/ the admixture of haloid acids into the atmosphere of sintering should not exceed 0.0001%, but this figure evidently would be revised. In our case the quantity of HCl has not been controlled, but decreases in weight in all tests and was constant. (3%)

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